MICROSTRUCTURE OF BINARY "METAL-SILICON" SYSTEMS TREATED BY LOW-ENERGY HIGH-CURRENT ELECTRON BEAMS

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Introduction

The action of quasi-stationary concentration energy flows is of a particular interest as a basis for the development of new methods for materials modification and formation of novel bulk and surface nanostructured composites /1-2/. Low-energy high-current electron beams (LEHCEB) is a specific type of these flows combining high energy density (5-40 J/cm^2) and controlled variable pulse duration (up to 200 μs). The use of LEHCEB for the synthesis and modification of silicon-based materials, especially metal silicides, has broad spectrum of applications in electronics, nanotechnology and new-generation nuclear power engineering /3-5/.

In this communication we discuss peculiarities of microstructural changes in "metal layer (Ti, Zr, Cr)-silicon substrate" systems treated by LEHCEB with energy density 8-15 J/cm^2 and pulse duration 50-200 μs. As it was shown in recent papers /2/, energy density of LEHCEB is sufficient for melting of the surface layer; therefore, mechanisms of structural transformation are mainly caused by solidification of the surface melt.

Experimental

Metal layers (Ti, Zr and Cr) were deposited on single-crystal silicon plate by vacuum arc deposition technique (cathodic arc current was 100 A, substrate bias – 120 V, deposition time 10 min). Thickness of the pre-deposited metal coating was 1,3-1,7 μm.

LEHCEB treatment was performed in a "Solo" installation developed in the Institute of High Current Electronics SB RAS /6/. The density of the energy transmitted to the target Q was 8-15 J/cm^2, pulse duration was varied within the range 50 to 200 μs. Treatment was carried out by a single pulse and a series of n=3 pulses with an interval of 1 s.

Surface morphology and microstructure of cross-section were studied by scanning electron microscopy (SEM) using a LEO1455VP microscope. Electron survey was carried out in phase contrast mode (registration of backscattered electrons only).

Results and discussion

SEM studies showed that LEHCEB action with energy density 8-10 J/cm^2 results in the formation of layered structure "metal-silicide-silicon" in Zr-Si system and formation of metal-rich dendrites in Ti-Si and Cr-Si systems (Fig. 1). Thickness of the interface silicide layer is 0,3-1,0 μm.

a) b)

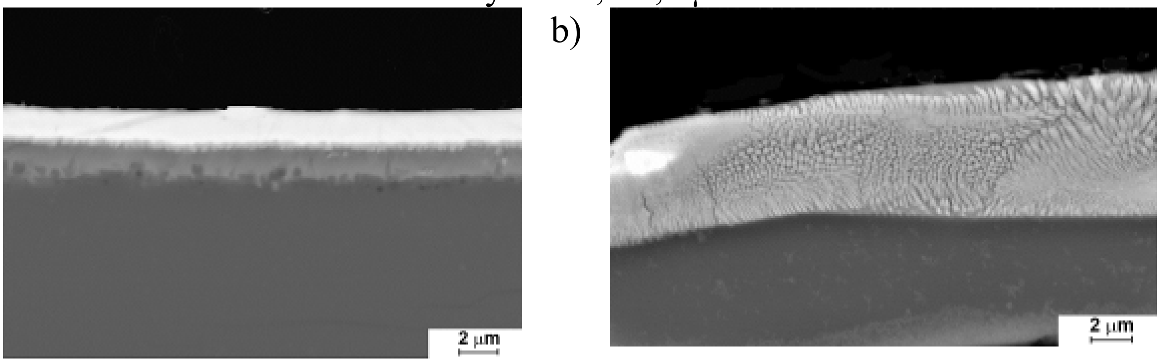
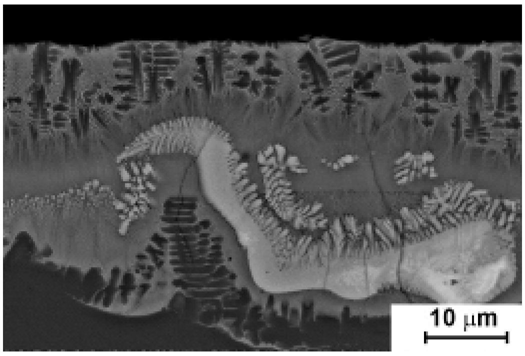


Fig. 1 Cross-section SEM-images of Zr-Si (a) and Cr-Si (b) systems treated by LEHCEB (n=1, Q=10 J/cm^2)

The formation of layered structure is caused by the melting of interfacial layer at "metal-silicon" interface when melting point of low-temperature eutectics is reached.

Dendritic growth in this case is caused by partial melting of the metal layer due to non-uniform lateral distribution of thermal energy density of the electron beam. In this case solidification starts at the boundary of the solid metal regions. Since temperature of the surrounding liquid is higher, temperature gradient at "solid-liquid" interface is directed from solid to liquid – i.e. it produces extra overcooling. This overcooling promotes the development of random excitations at the solidification interface that results in the growth of primary and secondary dendrite branches.

Increase of LEHCEB energy density results in the formation of deep intermixed layer (10-30 μm thickness) consisting of silicon dendrites (tip radius – 0,9 μm, primary dendritic spacing 16,8 μm, secondary dendritic spacing 1,5 μm), unmelt metal regions distributed throughout the alloyed layer and surrounded by metal-rich dendrites and nanosized eutectic (typical precipitates size is ~50 nm, eutectic grain size is 1-4 μm) (Fig. 2-3). Increase of LEHCEB results in the formation of two-layer structure (Fig. 2b). The upper layer consists of silicon dendrites and eutectics, in the underlying layer the unmelt metal is still present.

a) 

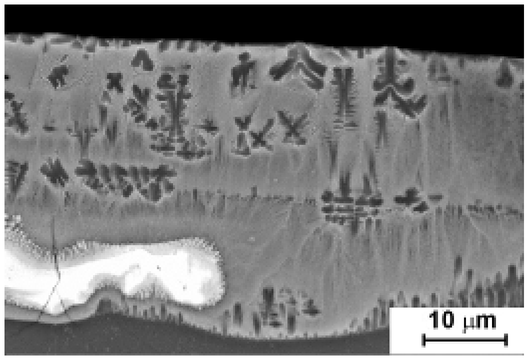
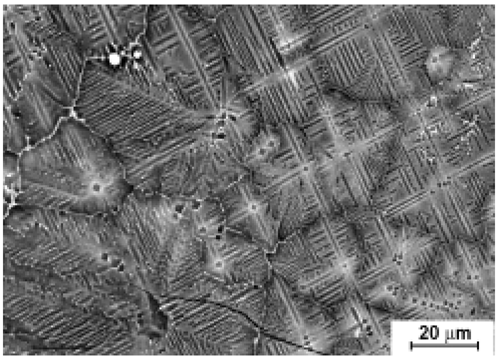
b) 

Fig. 2 Cross-section SEM-images of Ti-Si system treated by LEHCEB (Q=15 J/cm^2, n=1 (a), n=3 (b))

The formation of two-layer structure is due to the silicide growth. Since heat capacity of the silicon-silicide layer formed by the first LEHCEB pulse is less than that of metal and silicon, melting depth for next pulses is less and metal remains solid.

a) 

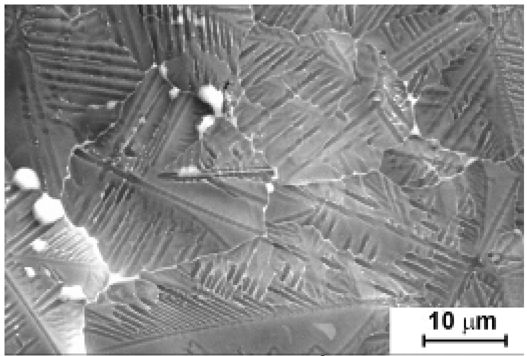
b) 

Fig. 3 Surface SEM-images of LEHCEB-treated Ti-Si (Q=15 J/cm^2) (a) and Zr-Si (Q=12 J/cm^2) systems

The formation of silicon dendrites is caused by low solid-state solubility of metals in silicon resulting in constitutional overcooling /7/. Silicon dendrites formation is caused by constitutional overcooling. According to phase diagrams /7/, solidification temperature of metal-rich liquid near the crystallization front is lower than that of silicon T_{Si}, i.e. liquid layer near the solidification interface is overcooled. In contrast with metal-rich dendrites, solidification of silicon is controlled by metal ratio gradient. The evolution of asperities near the crystallization front results in the formation of hexagonal cell and dendrites. Since metal is edged to the interdedritic space, the formation of metal silicides is most probable in these regions. When crystallization temperature of the intermixed melt reaches T_{Si}, further metal edging does not provide overcooling and dendrite growth stops.

Conclusions

The action of low-energy high-current election beams (LEHCEB) with energy density 8-10 J/cm^2 on "metal coating-silicon substrate" systems results in the formation of layered structure "metal-silicide-silicon" (Zr-Si system) and formation of metal-rich dendrites in (Ti-Si and Cr-Si systems). The formation of layered structure is caused by contact melting when melting point of low-temperature eutectics is reached. Dendrite growth is caused by partial melting of the metal layer due to non-uniform distribution of thermal energy density of the electron beam, metal melting and solidification with negative temperature gradient at "solid-liquid" interface. Increase of LEHCEB energy density results in the formation of deep intermixed layer (10-30 µm thickness) consisting of silicon dendrites (tip radius – 0,9 µm, primary dendritic spacing 16,8 µm, secondary dendritic spacing 1,5 µm) due constitutional overcooling at the solidification front.

Acknowledgments

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